

What is claimed is:

1. A process for producing an ammonium polythiomolybdate of the formula $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13} \bullet n \text{H}_2\text{O}$ where n is 0, 1 or 2 comprising:
 - (a) reacting an aqueous ammoniacal molybdate solution with hydrogen sulfide gas at superatmospheric pressure until the H_2S is no longer absorbed by the solution, said solution and said gas being in a closed system and the flow of said gas being regulated at an elevated pressure to form a slurry consisting essentially of a solid essentially all of which is ammonium tetrathiomolybdate containing a portion of the starting molybdenum and a mother liquor containing the balance of the molybdenum;
 - (b) heat soaking the reaction product of step (a) at elevated temperatures up to about 200°C in a closed reactor in the presence of elemental sulfur at a pressure of 600-1000 psig whereby the ammonium tetrathiomolybdate is converted to $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13} \bullet n \text{H}_2\text{O}$;
 - (c) cooling said slurry to ambient temperature;
 - (d) separating said solid from the major portion of said mother liquor;
 - (e) washing said solid with water followed by removing the resulting water washes to remove the remaining portion of said mother liquor and soluble impurities from said solid; and
 - (f) drying the resulting washed solid at ambient temperature to form the $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13} \bullet n \text{H}_2\text{O}$.
2. The process of claim 1 wherein the ammonium polythiomolybdate is $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13}$.

3. The process of claim 1 wherein the ammoniacal solution comprises MoO_3 , $(\text{NH}_4)_2\text{S}$ and elemental sulfur.
4. The process of claim 1 wherein the pressure in step (a) is 5-50 psig.
5. The process of claim 1 wherein the temperature in step (b) is 175-200°C.
6. The product prepared by the process of claim 1.